ACCELERATED TESTS OF ASPHALTS

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ABSTRACT

Equipment and methods for testing asphalt by accelerated weathering are described.

A variety of asphalts are shown to exhibit the same type of changes when exposed alternately to a cycle consisting of light from an inclosed carbon arc, water spray, and sudden temperature changes as when exposed outdoors under actual weather conditions.

CONTENTS

I. INTRODUCTION

This paper describes the equipment and methods developed at the National Bureau of Standards for accelerated weathering tests of asphalts; discusses the results obtained by its use; and suggests applications of this method in the solution of problems encountered in asphalt technology.

II. THE ACCELERATED WEATHERING CYCLE

The purpose of the accelerated weathering test is to produce rapid changes in the asphalt under conditions simulating outdoor exposure.

The equipment is essentially the same as that of Walker and Hickson,[2] consisting of three carbon arc lamps operating at 220 volts d. c. and 13 amperes. Each lamp is equipped with a cylinder with 3-inch slots for holding the test specimens or panels and each cylinder is geared to a driving mechanism which rotates it once in 20 minutes. Light spray simulating gentle rain is obtained from vertical water jets which may be operated in conjunction with the lights. Lawn sprinklers, with rotating arms, are used for vigorous spraying.

The heat produced by the lamp maintains a temperature of approximately 140° F. at the surface of the panels. A refrigerator, as a separate unit, is used to obtain sudden temperature changes.

Figure 1 gives a photographic view of the accelerated weathering equipment.

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[2] Accelerated Tests of Organic Protective Coatings, B. S. Jour. Research, **1**, p. 1; July, 1928.

The daily accelerated weathering cycle is as follows:

18 hours light (overnight).
1 hour refrigeration at −10° F.; three times per week.
3 hours rain (lawn sprinklers).

This cycle admits of numerous modifications to produce more frequent changes in the weathering conditions. However, in this work only the simplest cycle was used since, in general, more frequent changes do not affect the ultimate result, but merely hasten the test.

After the conclusion of the experiments described in this paper the cycle was modified as follows:

Cold, 1¾ hours, 8.45 a. m. to 10.30 a. m.
Rain, 1 hour, 10.45 a. m. to 11.45 a. m.
Light, 1½ hours, 12 m. to 1.30 p. m.
Rain, 2 hours, 1.45 p. m. to 3.45 p. m.
Light, 16½ hours, 4 p. m. to 8.30 a. m.

This cycle gives quicker results and is recommended as a tentative standard of test.

III. METHOD OF PREPARING TEST PANELS

For consistent results asphalt coatings of uniform thickness are essential. The following apparatus has given satisfactory results.

A brass pipe, 12 inches long and 1.5 inches in diameter, is mounted above a block of wood (2 by 4 by 12 inches); the frame being constructed to permit raising or lowering the pipe. An electric heating coil inside the pipe, controlled by a rheostat, heats the pipe to the desired temperature.

The asphalt is melted at the lowest possible temperature and poured on an aluminum panel and allowed to cool. The pipe is adjusted to give the thickness of coating desired and the panel is slowly passed back and forth between the pipe and block several times. If the panel is placed on several sheets of strong typewriter paper, folded once lengthwise, the upper layer of paper can be used to pull the panel back and forth and the lower paper will protect the block of wood from any excess asphalt. Care must be taken to keep the pipe clean. All coatings described in this paper were 0.025 ± 0.003 inch in thickness. Aluminum was used in these tests as a more suitable material for panels than glass, wood, or iron, because of freedom from breakage and relative freedom from moisture or air. Asphalt adheres very well to aluminum.

Panels for the tests in this paper were prepared by the simple device described above. An improved model of a coating machine for which the author is indebted to L. Kirschbraun, of the Flintkote Co., is shown in Figure 2. This improved model will be used in future tests.

IV. OUTDOOR EXPOSURES VERUS ACCELERATED TESTS

Preliminary outdoor exposures, to check results of accelerated tests, were made in Petri dishes, as this method of exposure is largely standardized in asphalt testing. Later the outdoor exposures were made on aluminum, prepared as for the accelerated tests.

The presence of cracks, extending through the coating to the metal, as evidenced by visual inspection, was taken as the end point

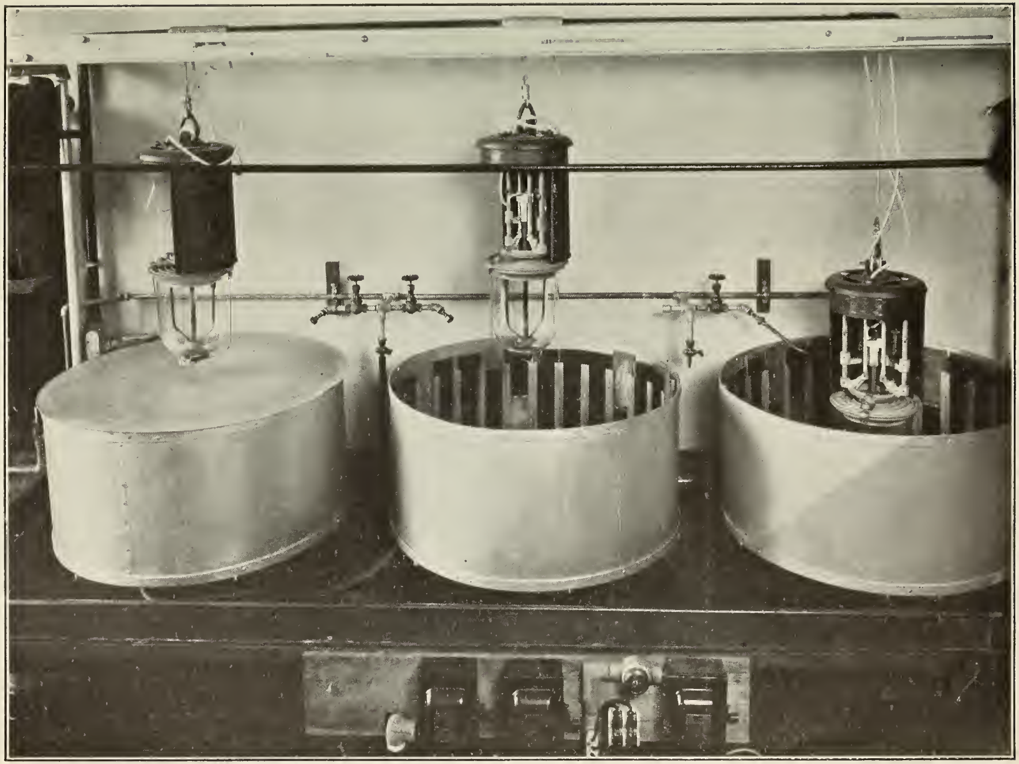

FIGURE 1.—*The accelerated weathering tester*

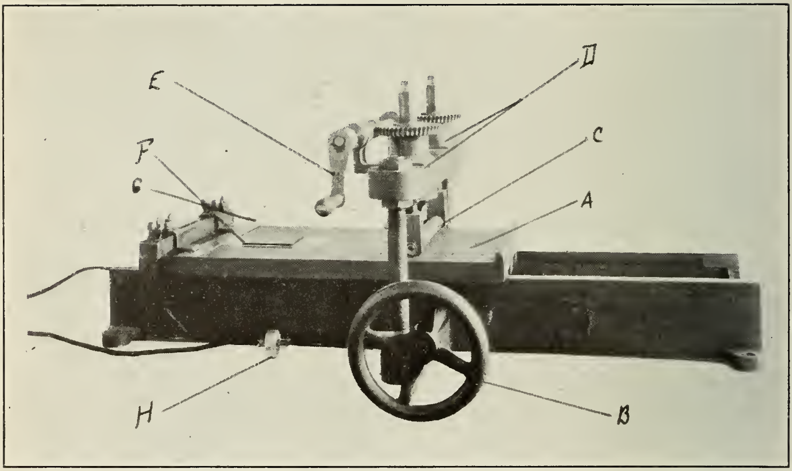

FIGURE 2.—*Coating machine with panel in position for coating*

A, Bed plate; *B*, hand wheel for moving bed plate; *C*, doctor, or heating tube (electrically heated); *D*, micrometer screws (graduated in 0.001 inch divisions); *E*, crank for turning micrometer screw and adjusting doctor; *F*, panel in position for coating; *G*, brass strip; *H*, plug and cord for heating element.

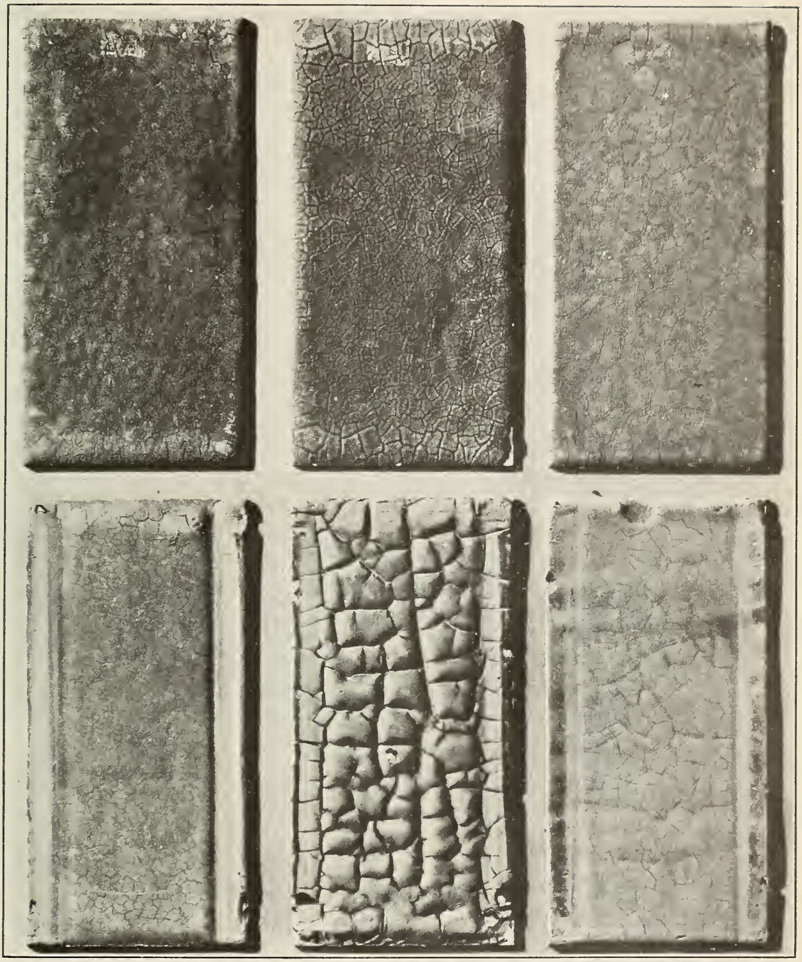

FIGURE 3.—*Outdoor versus accelerated exposure*

The panels shown in upper row were exposed outdoors to actual weather conditions for 1½ years. The panels immediately below these are their duplicates and were exposed in the accelerated test for 70 days.

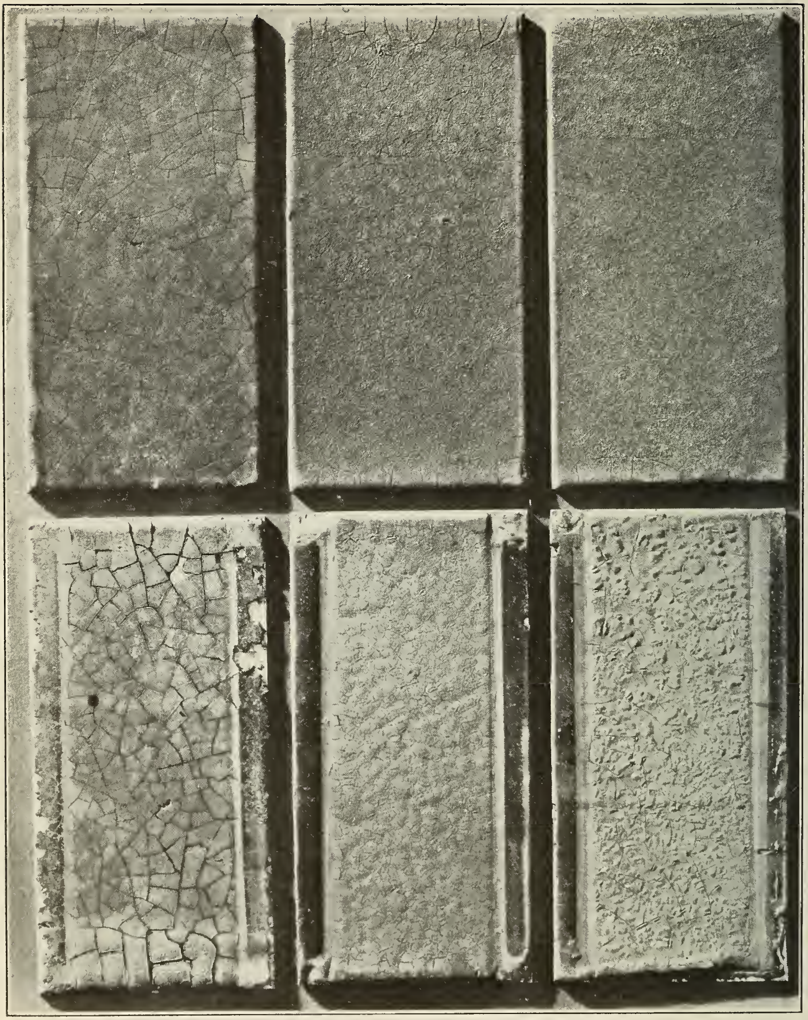

FIGURE 4.—*Outdoor versus accelerated exposure*

The panels shown in upper row were exposed outdoors to actual weather conditions for 1½ years. The panels immediately below these are their duplicates and were exposed in the accelerated test for 70 days.

of the test. In all cases the changes on outdoor exposure occurred in the same order as in the accelerated test, showing that the accelerated test is a true measure of the relative durability of asphalts.

In Figures 3, 4, and 5 the panels are especially grouped to bring out the relation between outdoor and accelerated weathering. The melting points of these asphalts were around 220° F. The panels were prepared by the method previously described by pouring the melted asphalt on slightly preheated aluminum sheet metal (3 by 6 by one-sixteenth inch) and then passing, after cooling, under the heated pipe until the desired thickness of 0.025 inch was obtained.

The panels shown in the upper rows of the photographs were exposed outdoors for one and one-half years. The asphalt samples exposed to the accelerated cycle are shown in the lower rows. These were exposed to the cycle for 70 days. An examination will show great similarity in appearance of these panels, particularly in the nature of their behavior as checking, cracking, flatness, and contraction.

In Figure 3 the first panel in the upper row shows similarity with its duplicate immediately below in its flatness and in the nature of its checks, which tend to form an irregular network. The word "flatness" refers to a peculiar smoothness of these panels. The center panels do not show flatness, but show a similar network of checks. The checking is more pronounced in the lower panel (accelerated test), but the type of change is the same. This sample was specially prepared to obtain an asphalt of poor weather-resisting qualities. The third panel (upper row) shows checking and straight cracking. The accelerated test (lower row) shows the same results as the outdoor exposure, only that the accelerated panel shows more flatness. The development of a "flat" and matt appearance is characteristic of paraffin-base asphalts.

In Figure 4 all the panels show flatness, most pronounced in the first and least in the center panels. The results obtained are similar in the accelerated and outdoor exposures. In the third set there are slight protuberances on the accelerated panel which do not show in the outdoor exposure. Refrigeration when used daily greatly retarded the formation of such protuberances, both as to size and number.

Figure 5 shows some new effects, more pronounced in the accelerated tests than in the outdoor exposures. In the third set of panels the checking is similar, as may more easily be detected by means of a magnifying glass. In the center set of panels grooving is shown, irregular in the outdoor panel and deep lines in the accelerated panel. The first set of panels show hummocks in addition to grooving, a mild form in the outdoor exposure and exaggerated in the accelerated exposure.

These examples have been given to show the remarkable similarity in the nature of the changes of asphalts under the two conditions of exposure. No attempt has been made to establish an exact ratio between outdoor exposure and the accelerated cycle.

V. EXPERIMENTS AND RESULTS OF ACCELERATED WEATHERING TESTS ON ASPHALTS

1. VALUE OF TESTS

The accelerated weathering cycle is valuable, not only because it produces in a short time results which are similar to long-time exposures outdoors, but also because it readily produces results which are useful in identifying kinds or types of asphalt and furnishes a method for detecting differences in the composition of asphalts. As Figures 3, 4, and 5 show, such types and differences of asphalts may also be detected upon outdoor exposure. The results, however, are not as distinct and the tests require much more time.

The results from the accelerated weathering tests may be described qualitatively and quantitatively.

The qualitative result is obtained in from 1 to 10 cycles of exposure to the accelerated cycle and is useful in determining the type of asphalt as evidenced by its characteristic behavior.

Asphalts may be grouped into types according to their characteristic behavior in the accelerated weathering cycle. In general, from four to six types should be distinguished for a complete study. For the sake of simplicity, however, only two types will be considered here, namely, type A and type B.

Type A.—These asphalts check when exposed to the accelerated cycle. They remain glossy for a relatively long time. After prolonged exposure to the cycle, the checks extend through the coating to the surface of the aluminum panel. Figure 6 illustrates this type of asphalt. This panel was exposed for 70 days to the accelerated cycle. The irregular form of the checks and their rounded edges (checking) are characteristic of this type of asphalt.

Type B.—These asphalts do not check. After 18 hours of exposure to light they become dull and show a flat, smooth, and streaked surface. When sprayed with water the asphalt takes on a grayish-green surface film. After prolonged exposure the asphalt cracks through to the surface of the aluminum. These cracks form long straight lines and run in every direction over the surface of the panel (cracking). This type of behavior is well shown in the first set of panels of Figure 4. This type is further illustrated by Figure 7. The asphalts in this group (fig. 7) were made from various kinds of crudes, but all came from the same petroleum fields. Their physical constants are shown in Table 1:

TABLE 1.—*Physical constants of asphalts*

Plate No.	Material	Softening point	Penetration	Ductility	Characteristic behavior to accelerated weathering	Remarks
		°F.				
100	Type B asphalt	223	25	1.4	Type B	These asphalts were made from various semiasphaltic crudes.
102	do	272	22	1.4	do	
106	do	230	25	1.8	do	
138	do	222	17	2.0	do	

When different asphalts are refined in the same manner it is frequently possible to determine their origin by exposure to the accelerated cycle and noting the characteristic behavior. However,

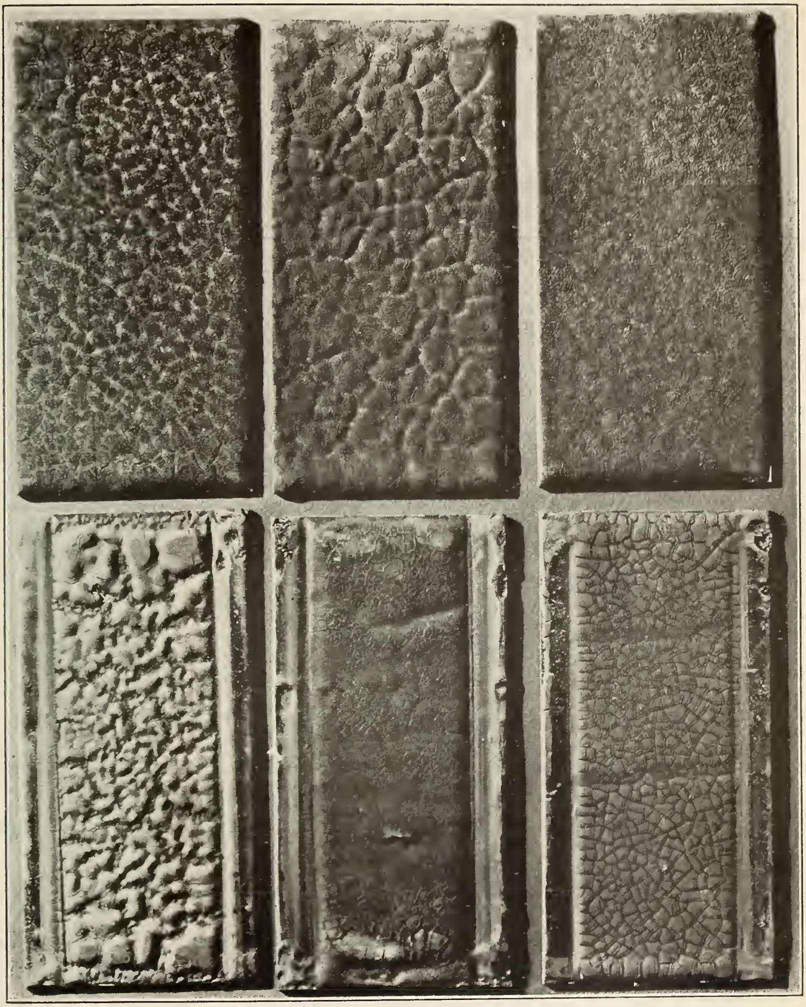

FIGURE 5.—*Outdoor versus accelerated exposure*

The panels shown in upper row were exposed outdoors to actual weather conditions for 1½ years. The panels immediately below these are their duplicates and were exposed in the accelerated test for 70 days.

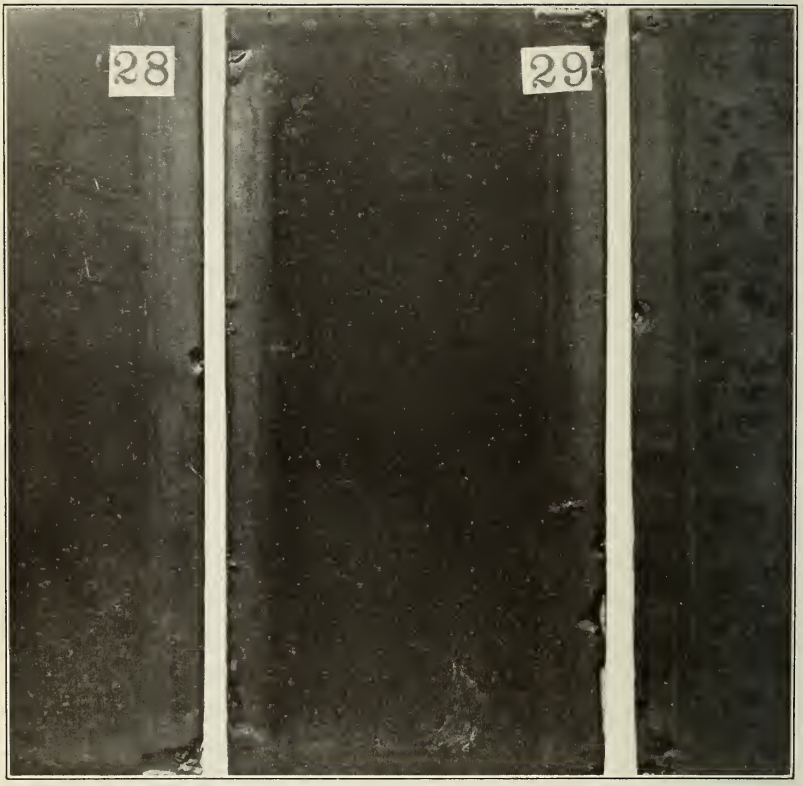

Figure 6.—*Accelerated exposure, type A asphalt*

The check formation is characteristic for this type of asphalt.

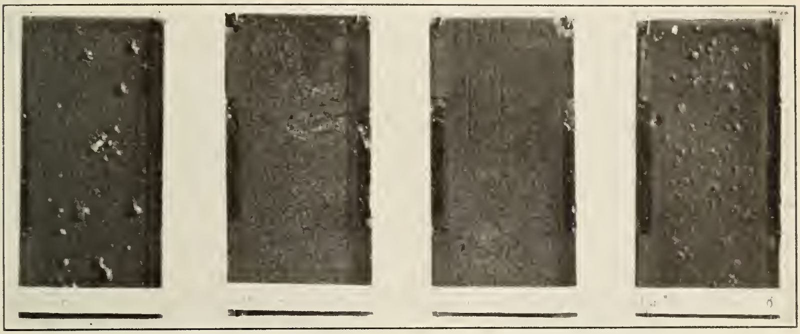

FIGURE 7.—*Accelerated exposure, type B asphalts*

The asphalts in this group were made from various kinds of crudes, but all came from the same petroleum fields. Their behavior is similar on exposure to the accelerated cycle. (See Table 1.)

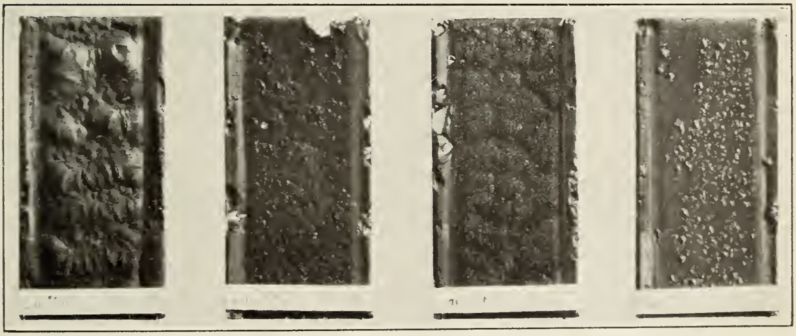

FIGURE 8.—*Accelerated tests*

Shows gradation of properties when asphalt fluxes are mixed before blowing. (See Table 2.)

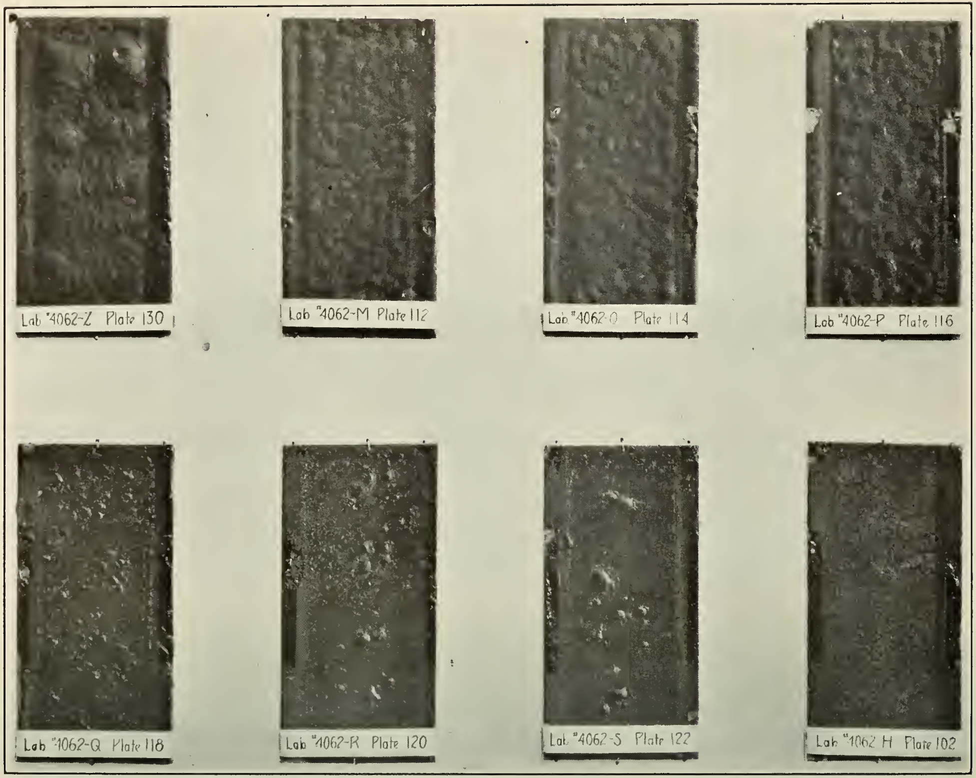

FIGURE 9.—*Accelerated tests*

These panels are mixtures of preoxidized asphalts. The asphalt present in greatest proportion determines the "type."

by changing the method of manufacture the crude oil can be made to produce asphalts that approach the appearance and behavior of other asphalts. In such cases it becomes difficult to detect the geographical origin of the crude oil from which the asphalt was made.

In Table 2 are listed the physical characteristics of mixtures of asphalts of the types A and B and their behavior in the accelerated cycle. These asphalts were prepared by mixing fluxes of type A and type B and then oxidizing them to the desired melting points. As seen in photographs of Figure 8, the behavior shows a gradation of properties from type A to type B.

TABLE 2.—*Physical constants of asphalts*

Plate No.	Material	Melting point	Penetration	Ductility	Characteristic behavior to accelerated weathering	Remarks
		°F.				
130	Type A	221	13	4.0	Type A (heavily grooved).	Shows gradation of properties from straight type A asphalt to straight type B. The fluxes were first mixed and then oxidized.
96	50 per cent type A flux; 50 per cent type B flux.	236	16	2.2	Combination of type A and B.	
92	70 per cent type B flux; 30 per cent type A flux.	217	16	2.6	do	
90	Type B	228	19	2.1	Type B	

In Table 3 the samples were prepared by mixing a flux of type A with a preoxidized asphalt of type B. Comparing panel No. 120 (fig. 9), with panel No. 92 (fig. 8) some differences in behavior are shown between mixtures of preoxidized fluxes and asphalts prepared by the oxidation of mixed fluxes. Panel No. 92 shows a behavior characteristic of a combination of types A and B, whereas the same percentage composition of A and B of panel No. 120 (but preoxidized) shows up as type B.

TABLE 3.—*Physical constants of asphalts*

Plate No	Material	Melting point	Penetration	Ductility	Characteristic behavior to accelerated weathering	Remarks
		°F.				
130	Type A	221	13	4.0	Type A (heavily grooved).	These samples are mixtures of preoxidized asphalts. The asphalt present in greatest proportion determines the "type."
112	20 per cent type B; 80 per cent type A.	216	15	3.6	Type A	
114	30 per cent type B; 70 per cent type A.	215	16	3.4	do	
116	40 per cent type B; 60 per cent type A.	214	18	3.3	do	
118	60 per cent type B; 40 per cent type A.	230	19	2.7	Type B	
120	70 per cent type B; 30 per cent type A.	235	19	2.3	do	
122	80 per cent type B; 20 per cent type A.	244	20	2.1	do	
102	Type B	272	22	1.4	do	

From this it would seem that oxidation of mixed fluxes produces an asphalt with characteristics of both fluxes, whereas in the case of mixtures of preoxidized asphalts the oxidized component present in greatest proportion determines the "type." This latter case is illustrated in Figure 9. Whether such behavior will always be found when mixing asphalts, only further investigation will show.

Some asphalts when exposed to the accelerated weathering cycle groove and contract. Similar results are obtained on outdoor exposures. (Compare center panels, fig. 3, and panel 130 of fig. 8.) In some cases this contraction is slight, in others pronounced. A pronounced case of this contraction and upheaval is shown in Figure 5, panel No. 1, lower row.

To determine whether the aluminum base caused this behavior, similar panels were prepared from invar metal, copper, pyrex glass, and saturated asphalt felt. Upon exposure to the cycle, the behavior in all cases was the same as on the aluminum.

To show that such contraction is due to the nature of the asphalt two samples were prepared in a small laboratory still. The crude was reduced for sample A to a specific gravity of 0.983 and for sample B to a specific gravity of 1.024. These residuums were then blown to a melting point of 200° F. In this manner two different asphalts prepared from the same petroleum base were obtained. Upon exposure to the accelerated cycle, sample B showed considerably more contraction than sample A. In Figure 5, the first panel of the lower row shows a pronounced case of such contraction as a result of the method of refining. In Figure 3, the center panels show contraction as a result of improper blending.

One of the important problems confronting the asphalt technologist is that of blending various asphalts to produce mixtures best adapted for the special purposes for which they are intended. This requires an intimate knowledge of the nature and behavior of the various materials and from the above experiments it would seem that such knowledge can best be acquired by use of the accelerated cycle.

The quantitative measure of the relative durability of an asphalt is regarded as the time required (usually 20 to 80 cycles) for the asphalt to crack down to the aluminum panel. If desired, the "electrical conductivity test" described by Walker and Hickson [3] may be used to determine cracking of the asphalt to the surface of the aluminum. For the present tests this method was not used, but the panels were studied by visual inspection which proved sufficient for these purposes.

Chemical analysis, according to the method of Marcusson,[4] was found to be useful in following the progress of deterioration. Asphalts consist of oils, resins, and asphaltenes, and upon weathering the content of oil and of resin decreases, whereas the asphaltene content increases.

In Table 4 the changes that take place in asphalts on weathering are indicated. Thus, the stage and rate of deterioration of asphalt may be determined by chemical analysis.

Table 4.—*Chemical analyses of asphalts*

Sample	Exposure to cycle	Asphaltenes	Resins	Oils
		Per cent	*Per cent*	*Per cent*
220° F. M. P. asphalt	None (original asphalt)	26.2	4.8	68.0
	21 days	34.6	1.2	61.4
	80 days	40.6	1.2	56.6

[3] See footnote 2, p. 247.
[4] Herbert Abraham, Asphalts and Allied Substances, 3d ed., p. 755.

The value of such chemical analysis is the greater in that it will permit a definite comparison between outdoor and accelerated weathering.

VI. SUMMARY

The experiments have demonstrated that similar results are obtained when asphalts are exposed to the accelerated cycle and to outdoor weathering.

It was found that asphalts show a characteristic behavior when exposed to the accelerated cycle, so that it is possible to classify the asphalts into "types of behavior."

It was further shown that some grades of asphalts contract when exposed to the cycle, as well as outdoors.

The value of the chemical analysis of asphalts to determine progress of change was indicated.

WASHINGTON, February 1, 1930.